

# Sol–gel approach to near-net-shape oxide–oxide composites reinforced with short alumina fibres—The effect of crystallization

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## Abstract

Near-net-shape (NNS) high alumina (alumina:silica = 96:4, in equivalent weight ratio) fibre reinforced ceramic matrix composites (CMCs) were prepared with single and bicomponent sols following sol–gel vacuum infiltration technique. The CMCs were characterized by X-ray diffraction (XRD), three-point bend test and scanning electron microscopy (SEM). Crystallization of tetragonal zirconia (t-ZrO<sub>2</sub>) in the composite, CZY having zirconia–yttria matrix and that of gamma alumina ( $\gamma$ -Al<sub>2</sub>O<sub>3</sub>) in the composites, CAZ having alumina–zirconia matrix, CAS having alumina–silica matrix and CA having alumina matrix, enhanced the flexural strength values and pseudo-ductile character of CMCs.

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## 1. Introduction

The use of monolithic ceramics has some restrictions in structural applications because of their inherent catastrophic nature. The development of ceramic fibre reinforced ceramic matrix composites (CMCs) has been proved to be promising alternative to monolithic ceramics for structural materials fabrication in engineering field [1,2]. If CMCs could be made consistently and at reasonable cost, they could be ideal for high temperature corrosive and abrasive environments. One of the processes of fabrication of CMCs is to infiltrate a liquid matrix into a reinforcement assemblage of required shape and size which is called preform [2]. These preforms may be continuous or discontinuous fibres or whiskers or particulates assemblages prepared to the desired volume fraction, shapes and sizes [2,3].

Several techniques are currently used to fabricate CMCs with NNS capability which is used to produce NNS components that do not require machining or working with diamond tools. The conventional methods of producing CMCs are hot pressing [4], melt infiltration [5], chemical vapour infiltration (CVI) [6], slurry infiltration-high pressure sintering (SI-HPS) [3,7,8] and sol–gel

[3,8–14]. Radsick et al. [8] fabricated oxide/oxide matrix composites by slurry infiltration-hot pressing sintering technique (SI-HP). Recently Ruggles-Wrenn et al. [14] have studied the environmental effects on the creep behaviours of oxide/oxide ceramic matrix composites prepared by sol–gel technique. Dey et al. [11] have fabricated near-net-shape fibre reinforced ceramic matrix composites by sol infiltration technique. Sol–gel vacuum infiltration technique is a promising route for the fabrication of oxide–oxide ceramic matrix composite. In the vacuum infiltration technique, a solution containing metal compounds, e.g., metal alkoxides, acetates, nitrates or halides are treated chemically to form a sol which is then infiltrated into the fibre preform. The sol in the preform is converted to gel form by physical and/or chemical means, which in turn is subjected to control heating to produce the desired products e.g., glass, glass–ceramic or ceramic matrix. The main advantages of this technique are lower processing temperature and higher compositional homogeneity with respect to slurry infiltration-hot pressing sintering technique (SI-HP). In this process, preforms provide a uniform distribution of fibres and a very high surface area to the matrix gel. However, during drying, shrinkage of matrix results in crack formation in the matrix. It can be minimized by repeated infiltrations with the sol matrix and subsequent drying.

The properties of CMCs are influenced by the characteristics of the reinforcing fibres i.e., their strength, aspect ratio,

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chemistry and thermal stability [2,15]. The use of sintering aid also influenced the mechanical strength of the CMC [16]. For the development of a high quality CMC, matrix properties are also to be taken into account. Important characteristics of the matrix include thermal compatibility with the fibres and fibre–matrix interface reaction. The strength and toughness of CMC are primarily governed by the interfacial bonding at the fibre/matrix interface [1,15]. Interfacial strength can be influenced by a number of factors, most important being degree of bonding between the fibre and the matrix and their difference in thermal expansion behaviour.

Crystallization behaviour i.e., the development of crystalline phases in the composites with sintering temperatures has an important role for the characteristics of the CMCs. In the present investigation, a sol–gel vacuum infiltration route was followed for the preparation of NNS ceramic fibre reinforced ceramic matrix composites using various sols as the infiltrates. The effect of crystallization in the composites towards their characteristics i.e., mechanical strength, pseudo-ductility and hence the nature of fibre–matrix interface has been studied in this work.

## 2. Experimental procedure

### 2.1. Preparation of precursor sols

Zirconium oxychloride octahydrate,  $ZrOCl_2 \cdot 8H_2O$  (Indian Rare Earths Ltd., Mumbai, India, purity > 99%), hydrated yttrium nitrate (Indian Rare Earths Ltd., Mumbai, India, purity > 99%), aluminum nitrate,  $Al(NO_3)_3 \cdot 9H_2O$  (G.R. Merck, Mumbai, India, purity > 99%) and tetraethylorthosilicate, TEOS (Fluka Chemie AG, Switzerland, purity > 98%) were used as the starting materials for  $ZrO_2$ ,  $Y_2O_3$ ,  $Al_2O_3$  and  $SiO_2$  respectively. Fig. 1 shows schematically the preparative steps of zirconia–yttria (ZY), alumina–zirconia (AZ), alumina (A) and alumina–silica (AS) sols.

For the preparation of ZY sol with the composition  $94ZrO_2 \cdot 6Y_2O_3$  (in equivalent mole),  $ZrOCl_2 \cdot 8H_2O$  (1.5 M) was precipitated as hydrated zirconia by the addition of aqueous ammonia solution (25 wt.%, G.R. Merck, Mumbai, India) under stirring, maintaining the pH of the solution up to

8–9. The precipitate was washed with deionized water to free foreign ions e.g.,  $Cl^-$ ,  $NH_4^+$  etc. followed by peptization with glacial acetic acid (99.8%, AnalaR, BDH, Mumbai, India) at  $65 \pm 1^\circ C$ . A required amount of  $Y(NO_3)_3$  (6 mol% equivalent  $Y_2O_3$ ) solution was added to the above peptized zirconia sol under stirring for 15 min at r.t ( $30^\circ C$ ) to prepare ZY sol (Fig. 1).

For the preparation of AZ sol of composition  $87Al_2O_3 \cdot 13ZrO_2$  (in equivalent mole), required amount of  $Al(NO_3)_3$  solution (1.5 M) was mixed with required amount of  $ZrOCl_2$  solution (1.5 M) under stirring for 15 min. An aqueous ammonia solution (25 wt.%, G.R. Merck, Mumbai, India) was added to the above mixed solution containing  $Al^{3+}$  and  $Zr^{4+}$  under stirring at  $80 \pm 1^\circ C$  in a covered container (Fig. 1). In this case the adjustment of pH at around 3 is the crucial step for stabilizing of AZ sol.

For the preparation of alumina sol (A), ammonia solution (25 wt.%, G.R. Merck, Mumbai, India) was slowly added to 1.5 M of  $Al(NO_3)_3$  solution at  $80 \pm 1^\circ C$  under stirring in a covered container. The pH of the sol (A) was then adjusted up to about 3 by control addition of  $NH_4OH$  in warm condition (Fig. 1).

A calculated quantity TEOS was slowly added under stirring to the required amount of alumina sol (A) as prepared above under stirring to obtain AS sol of composition  $60Al_2O_3 \cdot 40SiO_2$  (in equivalent mole). The stirring was continued for 15 min for homogenization (Fig. 1).

The viscosity values of the above sols (ZY, AZ, A and AS) were adjusted up to either 10 or 5 mPa s by solvent evaporation. Two series of sols were prepared with different viscosities: one with a viscosity of 10 mPa s for the very first infiltration steps and the other with a viscosity of 5 mPa s for the last infiltration step(s).

### 2.2. Infiltration of the sols in the high alumina fibre preforms

In the present investigation, the high alumina fibre preform (M/s Thermal Ceramics, USA) (fibre content = 30 vol.%, length = 200–250  $\mu m$ , diameter = 3–7  $\mu m$ ) of dimension 125 mm (diameter)  $\times$  25 mm (thickness) was infiltrated with the above prepared sols, ZY, AZ, AS and A using a custom-designed set-up (Fig. 2). In this technique, the fibre preform was placed on a filter bed (sintered disc) of the infiltration unit with the help of a specimen holder. The preform was immersed with the prepared sol of viscosity 10 mPa s by sucking it up through the filter bed with the help of a rotary vacuum pump attached to the infiltration unit. The samples were kept for 5 min in the sol under immersed condition followed by releasing pressure. The sol infiltrated preform was dried at  $100^\circ C$  to convert the penetrated sol into the corresponding wet gel. This process was continued thrice with the same sol of viscosity 10 mPa s. The wet infiltrated preform was then heated at  $400^\circ C$  in air under static condition to remove the volatiles and decomposable materials. Finally, the  $400^\circ C$ -treated sample was infiltrated following the above procedure with the sol of viscosity 5 mPa s followed by heating at different temperatures, i.e., 800–1400  $^\circ C$  in air under static condition.

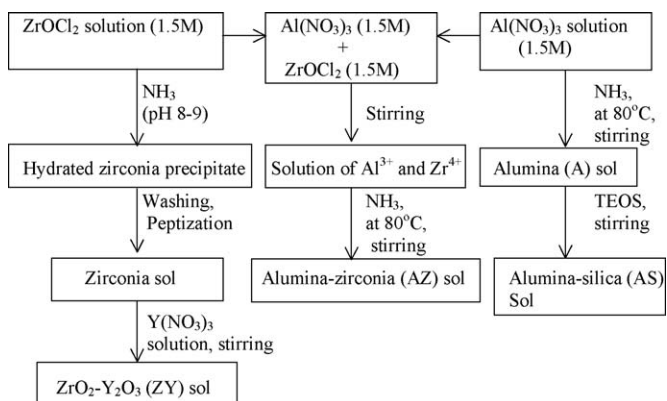


Fig. 1. Schematic for the preparation of zirconia–yttria (ZY), alumina–zirconia (AZ), alumina (A) and alumina–silica (AS) sols.

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